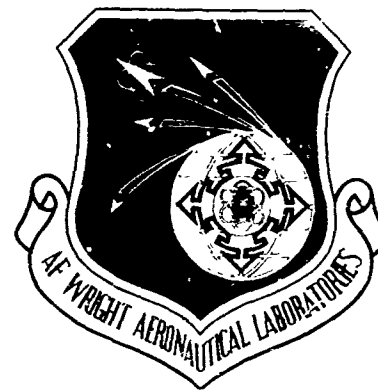AD-A197 109

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BORIC OXIDE AS A SOLID LUBRICANT ADDITIVE
IN MOLYBDENUM DISULFIDE

CHRIS J. KLENKE
Lubrication Branch
Fuels and Lubrication Division

MARCH 1988

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A recent investigation suggests that antimony trioxide (Sb_2O_3) performs well as an additive in molybdenum disulfide (MoS_2) because of its ability to soften at asperity contacts so that the solid lubricant can attain and retain a preferred tribological orientation.

This research determined the effectiveness of boric oxide (B_2O_3), when used as an additive in MoS_2, for substrate temperatures ranging from 21°C to 316°C. This range was used to allow the asperity contact temperature to vary below and above the softening point of B_2O_3. It was found that a moderate friction coefficient and high wear, which is attributed to the additive acting abrasively, occurred when the asperity contact temperature was well below the softening point of the oxide. When the asperity contact temperature neared the softening point of the oxide, the friction coefficient increased dramatically and the wear volume was reduced. It is postulated that B_2O_3 acted adhesively at the interface resulting in a higher coefficient of friction and wear decreased due to an attainment of a preferred

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orientation by the MoS_2. For asperity contact temperatures significantly above the softening point of B_2O_3, the friction coefficient returned to about the same value as for temperatures below the softening point. It is speculated that B_2O_3 liquifies resulting in the oxide acting less adhesively thereby lowering the friction coefficient. This liquification also provides a mechanism for greater loss of MoS_2 and an increased wear rate.

These observations support a hypothesis that an additive. such as boric oxide, can soften as the asperity contact temperature approaches the softening point temperature of the additive so that the overall tribological conditions may be improved resulting in reduced interfacial wear. Different thermal or pressure-velocity (PV) conditions, for this same additive, will significantly detract from optimum conditions.

PREFACE

This technical report was prepared for the Lubrication Branch, Fuels and Lubrication Division, Aero Propulsion Laboratory (APL), Air Force Wright Aeronautical Laboratories (AFWAL), Air Force Systems Command, Wright-Patterson Air Force Base, Ohio. The work herein was accomplished under Project 3048, Task 304806, Work Unit 30480626, "Turbine Engine Lubricant Research" for the period January 1987 to June 1987, with Mr Chris J. Klenke, AFWAL/POSL, as project engineer.

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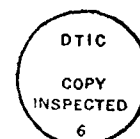
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SECTION I

INTRODUCTION

The limit at which synthetic fluids can act as a lubricant is generally accepted to be around 700°F due to vaporization and thermal degradation (1). At this, and higher temperatures, solid lubricants become very important due to their thermal and oxidative stability. Solid lubricants are also important for other applications in which liquid lubricants can become contaminated or cannot support the required load (2). Other advantages of solid lubricants are good stabilities at extreme temperatures, light weight, and better separation of mating surfaces when sliding slowly under high loads. Some disadvantages of a solid lubrication system include the following: friction coefficients are generally higher for solid lubricants than for fluids, finite wear lives, and feed rates of gas - entrained solid lubricants must be carefully controlled to prevent clogging and binding with excess lubricant if a powder delivery system is employed (3). Examples of solid lubricants in use today are molybdenum disulfide (MoS_2) and graphite.

Various materials may be added to solid lubricants for the purpose of improving the wear volume and/or friction coefficient. In order to narrow down the list of candidates for a solid lubricant additive, it is important to understand how the additive performs beneficially and the criteria which are critical in their selection.

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Recent research presents a hypothesis which states that some additives improve solid lubricant performance due to the additives' ability to soften at the asperity contacts, under certain conditions, which permits the solid lubricant to attain and retain a preferred orientation thereby improving the overall tribological situation (4). The number of real area contacts between the pellet and substrate, at any one time, is highly dynamic. Pictures taken of steel pins sliding on a disk, with exposure times of less than 33 milliseconds, show the number of asperity contacts to be roughly constant at seven plus or minus three, suggesting a minimum three point contact. The asperity contact temperature is estimated to be roughly 200°C above the surface temperature of the substrate (5).

To more closely examine this hypothesis boric oxide, B_2O_3, was selected for formulation with MoS_2 so that tribological studies could be conducted above and below the glass transition (T_g) temperature (253°C) of the additive to determine where performance of the solid lubricant would be enhanced by the additive. Two criteria used to assess the performance of MoS_2/B_2O_3 formulations were the friction coefficient and wear volume in a sliding wear test.

SECTION II

EXPERIMENTAL

To assure that wear volumes and friction coefficients generated in the pellet tests were not due to a few, large, hard particles, the B_2O_3 was sieved through a nickel screen to a size no larger than 75 microns (Fig. 1). 53.4 mg of the sieved B_2O_3 and 246.4 mg of MoS_2 were mechanically mixed in a test tube with a vortex spinner to insure good dispersion of the two powders. The mixed powder was formed at about 25000 psi into a compact cylinder with a conically shaped tip. The cone was then trimmed on a lathe to an angle of 21.8 degrees (Fig. 2). The compressed pellet was mounted and counterbalanced in a MRI MARK VB wear test machine (6) and axially loaded with 100 grams. The cone rested on an isopropanol washed, 0.002 inch thick, stainless steel substrate which was rigidly mounted on a heated metal support. The substrate was rotated at 450 rpm for each test. Insignificant, minor variations in rpm was noted throughout the tests. The pellet arm holder is linked to a linear voltage differential transformer (LVDT). The tangential load exerted by the sliding pellet causes a deflection of the LVDT which was recorded continuously throughout each test on a strip chart recorder. LVDT deflection, as a function of static load, was calibrated before each run. The distance between the cone tip and the center of rotation was measured after each run. Friction coefficients were calculated using these data.

The wear test machine is equipped with a bell jar permitting control of the test atmosphere. Before each run, a vacuum was drawn to 10^{-3} torr and the chamber then recharged with filtered, dry air. This process was
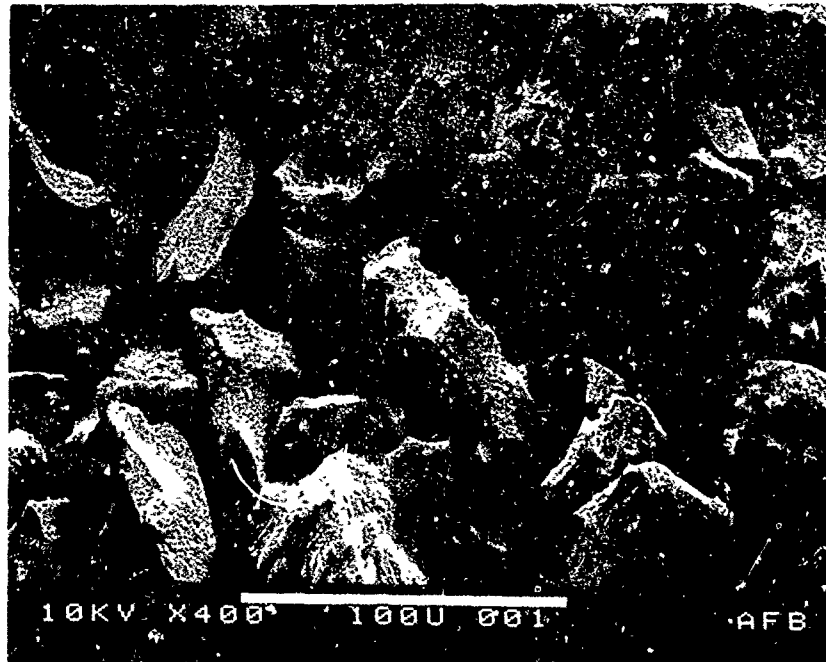
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Figure 1. Sieved Boric Oxide

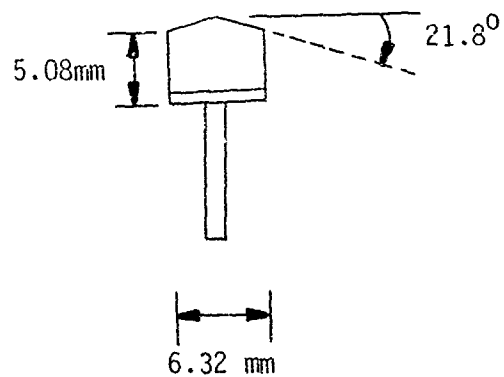
Figure 2. Wear Pellet Configuration

repeated three times to insure a dry atmosphere. B_2O_3, MoS_2, and compacted pellets were stored in a dessicator at all times other than during preparation or testing.

Triplicate pellet tests were run for 10 minutes at 21°C. 100°C, 150°C, 200°C, 250°C, 300°C, and 316°C. After each test, the diameter of the wear cone was measured using a Baush and Lomb stereomicroscope which was calibrated using a stage micrometer. The wear volume of each pellet was calculated knowing the radius of the wear cone and the geometric configuration of the pellets. Calculation of wear volume is as follows:

$$\text{Volume} = (\pi/3)r^2h = 1.047r^2h$$

for a 21.8° cone, $h = 0.39997r$

or, volume $= 0.4188r^3$

where r = radius of wear cone base

and h = height of wear cone of removed material

After each test, the worn pellets were evaporatively coated with gold and the wear area examined in an Amray 1600 Scanning Electron Microscope (SEM).

SECTION III

RESULTS

Summaries of the friction coefficients and wear volumes as a function of test temperatures are given in Tables 1 and 2. Outlying data were omitted for various reasons including failure to vent the chamber to air before a run and breaking of the conical pellet tip at the start up of a test.

As seen, the data indicate that the friction coefficients increase significantly up to 150°C after which there is a dramatic decline. The average wear volume is large at 21°C with a value of 0.42 mm^3. Reduced wear volumes are generated up to a test temperature of 150°C where 0.02 mm^3 of wear is generated. As the temperature is increased to 315°C, the wear volume increases slightly to 0.06 mm^3.

SEM photographs of the room temperature wear pellets exhibited voids in the wear surfaces (Fig. 3). Appearance of the wear surfaces for tests ran below 150°C are irregular with jagged edges. At the test temperature of 150°C, the wear surface became more circular with rounded edges. Evidence of plastic deformation is observed in the wear surface for pellets ran at a test temperature of 150°C (Fig. 4). As temperatures are further increased to 316°C, the wear surface retains a rounded appearance with a smooth surface. Plastic deformation of material, in the wear areas, is still evident at 316°C.

TABLE 1

AVERAGE FRICTION COEFFICIENT VERSUS TIME AND TEMPERATURE

TEMPERATURE (°C)	1 MINUTE	5 MINUTES	10 MINUTES
	0.196 ± 0.004	0.191 ± 0.029	0.173 ± 0.030
	0.191 ± 0.049	0.157 ± 0.007	0.146 ± 0.007
	0.251 ± 0.012	0.285 ± 0.020	0.233 ± 0.011
	0.152 ± 0.046	0.184 ± 0.028	0.185 ± 0.059
	0.155 ± 0.024	0.205 ± 0.053	0.176 ± 0.066
	0.158 ± 0.025	0.158 ± 0.031	0.153 ± 0.049
	0.196 ± 0.050	0.192 ± 0.057	0.122 ± 0.058

TABLE 2

AVERAGE WEAR VOLUME VERSUS TEMPERATURE

TEST TEMPERATURE (°C)	WEAR VOLUME (mm³)
25	0.425 ± 0.060
100	0.039 ± 0.003
150	0.018 ± 0.001
200	0.033 ± 0.006
250	0.039 ± 0.012
300	0.057 ± 0.031
355	0.051 ± 0.017

Figure 3. Wear Surface Containing Voids at 21°C

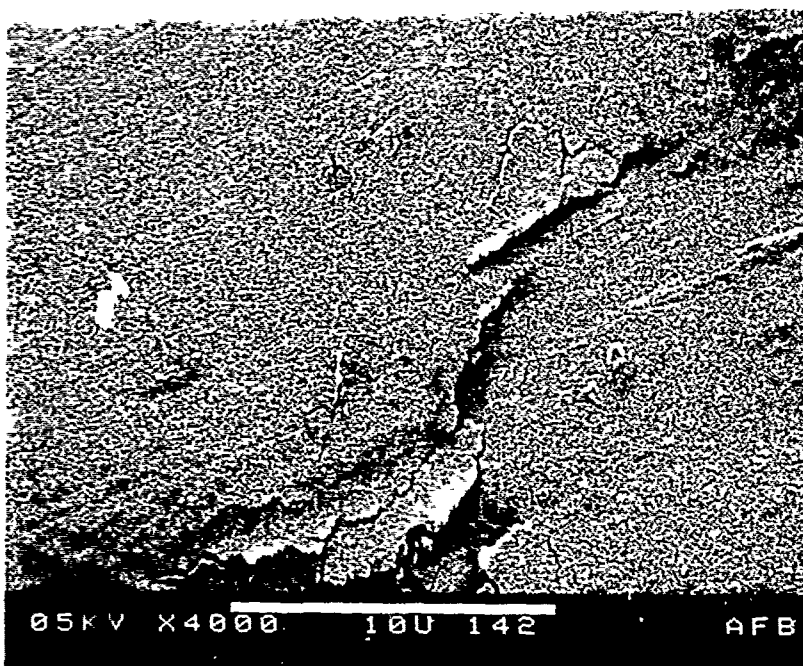Figure 4. Wear Surface Exhibiting Areas of Plastic Deformation at 150°C

SECTION IV

DISCUSSION

The wear data generated indicate that wear volume for B_2O_3/MoS_2 compacts varies with test temperature. Wear volumes of the pellets ran at room temperature were large compared to wear generated at higher temperatures. A SEM examination of the 21°C wear surface revealed many voids (Fig. 3). It is speculated that B_2O_3 acts as an abrasive at the interface and generated voids when some of the oxide particles are torn from the compact matrix. This mechanism explains the high wear volume occurring at room temperature. The inclusion of brittle, abrasive material into the MoS_2 would also account for the comparatively high friction coefficient.

At the test temperature of 150°C, there is a noticeable reduction in the wear volume and increase in friction coefficient as compared to the lower test temperatures (Figs. 5 and 6). The low wear volume at this temperature is attributed to B_2O_3 softening at asperity contacts and plastically deforming, thereby allowing MoS_2 crystallites to obtain and retain a preferred tribological orientation.

The increase in the friction coefficient, at 150°C, may be rationalized by an increased "tackiness" of the B_2O_3, which is now acting adhesively. The result of this increased "tackiness" is the creation of a large tangential force resulting in an increased friction force (Fig. 6). Similar behavior has been observed for boron carbide sliding on stainless steel in air. In that system, boron carbide oxidizes to form a "sticky" boron oxide film which then increases the friction (7).
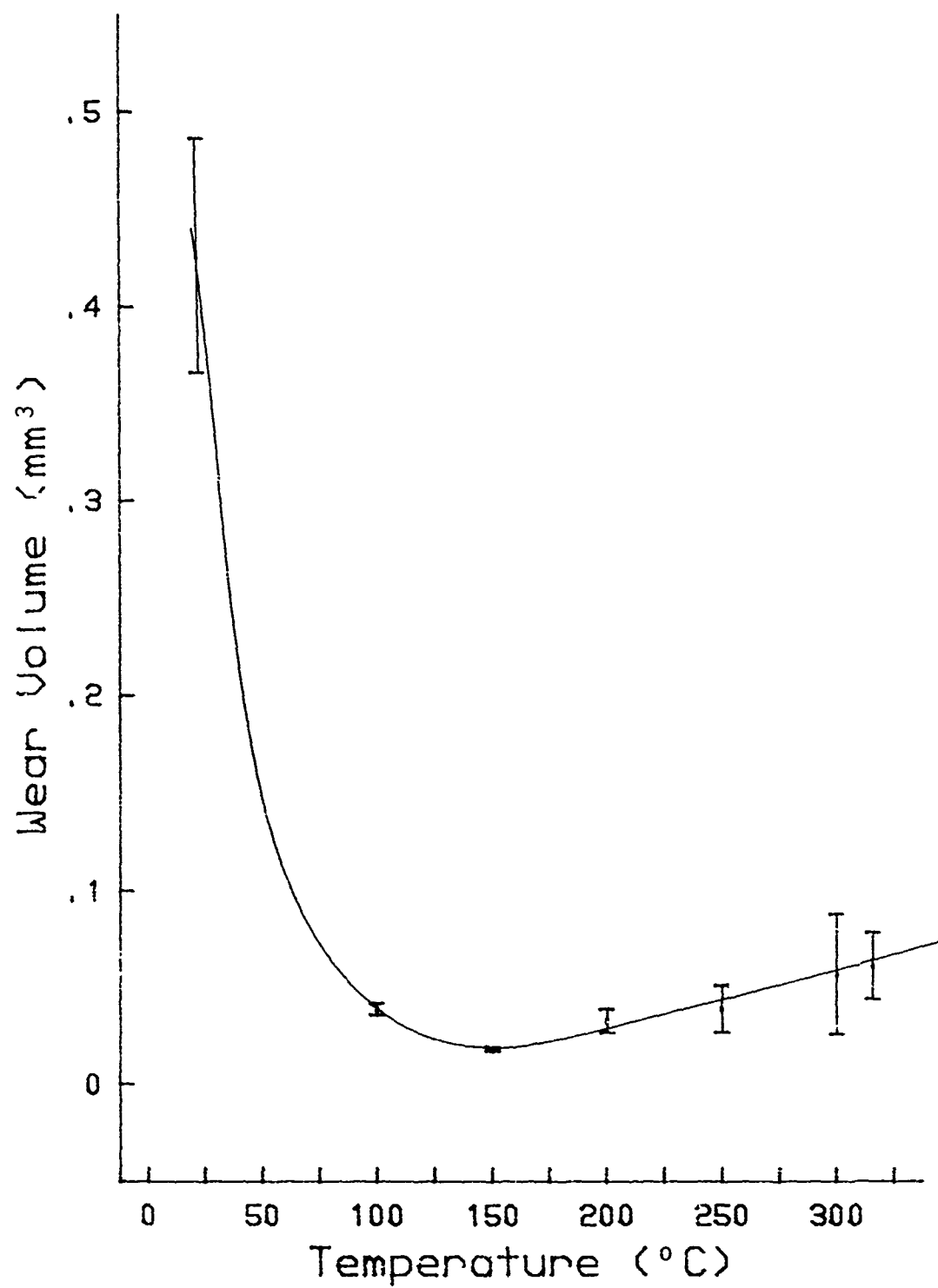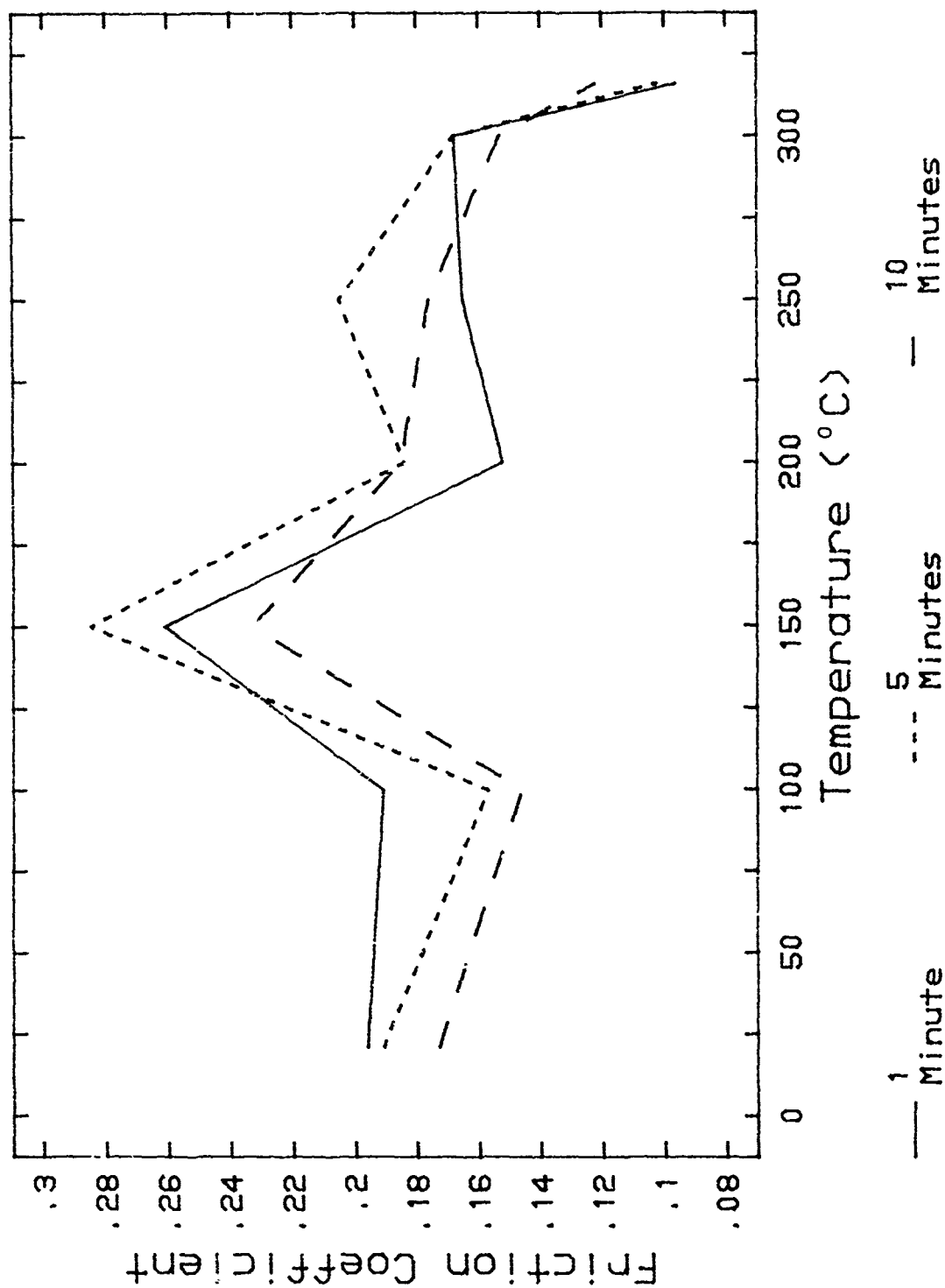
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Figure 5. Effect of Temperature on Wear Volume

Figure 6. Effect of Time and Temperature on the Friction Coefficient

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It has been shown that the asperity contact temperature for sliding speeds in the range of 200 cm/sec with selected materials is generally about 200°C above the test temperature (8). There are, however, many other factors which can influence the actual temperature of these contact areas. For these series of tests, the sliding speed was 50 cm/sec which would create lower asperity contact temperatures than the predicted estimate because less frictional heat is being generated and contact materials are different. The softening point of the B_2O_3, which corresponds to its T_g, has been reported as 253°C (526°K) (9). It would not be unreasonable, therefore, to assume that the asperity contact temperature would be in the range of 250°C to 300°C, at substrate temperature of 150°C. It should be kept in mind that water may play a role in determining the T_g of B_2O_3 which in turn will effect the temperature at which B_2O_3 will soften. The T_g of a substance, such as B_2O_3, is also, of course, a dynamic quantity which depends on the rate of cooling or heating of the amorphous material which may influence near surface performance of the additive.

When the substrate temperatures were increased to above 150°C, the wear volume increased slightly and yielded a friction coefficient equal to the average value it possessed below 150°C. The fact that the frictional coefficient returned to its pre-150°C value lends support to the concept that the high friction coefficient at 150°C is caused by the B_2O_3 becoming tacky.

At temperatures higher than 150°C, it is presumed that B_2O_3 becomes much less viscous at the asperity contacts which permits MoS_2 to be removed from the interface, which provides a rationalization for a lower coefficient of friction and higher wear rate.

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Data from a previous study (4) show that the wear volume for MoS_2 is around 0.2 mm³ for a 20 minute run at 21°C. B_2O_3/MoS_2 run under the same conditions for only 10 minutes, generated a wear volume of 0.43 mm³. B_2O_3/MoS_2 compacts tested at temperatures over 100°C, generated average wear volumes under 0.06 mm³ while MoS_2 alone exhibited wear volumes over 0.17 mm³. Friction coefficients for B_2O_3/MoS_2 pellets at those temperatures, with the exception of 150°C, were around 0.15 which are similar to those generated by MoS_2.

The wear data suggest that there is a test temperature at which there will be a minimum wear volume (Fig. 5), due to complex physical interactions between the solid lubricant, additive, and the substrate surface, which corresponds to near the maximum in friction coefficient (Fig. 6).

SECTION V

CONCLUSIONS

This study supports the hypothesis that selected additives soften under appropriate asperity contact conditions permitting the solid lubricant to attain and retain a preferred orientation thereby improving tribological conditions. B_2O_3 used here as an additive in MoS_2 resulted in a maximum 64% reduction in wear volume and an increase in the friction coefficient when the asperity contact temperature nears the softening point of the oxide.

Test temperatures significantly below the softening point of the oxide evidently causes the additive to act as an abrasive with high wear and a high friction coefficient. At relative high temperatures, B_2O_3 becomes softer, perhaps even to the point of becoming liquid, permitting easy removal of compact material at or near the contact points. Thus, the increased fluidity of B_2O_3 causes it to be less adhesive which lowers the friction coefficient but also provides a mechanism for greater loss of MoS_2 and increased wear rate.

The study also supports a concept that a solid lubricant additive or a lubricant by itself, that acts benefically due to softening, is likely to have a small envelope of usefulness in terms of temperature, pressure, and sliding speeds. This is in contrast to materials such as MoS_2 and graphite, which act as lubricants because of their laminar structure and have a much larger envelope of usefulness in terms of temperature, pressure, and sliding speeds.

REFERENCES

1. Peterson, M. B., "High Temperature Lubrication," <u>Proceedings from Lubrication and Wear</u>, D. Munster and B. Sernlicnt, Eds., McCutchan Publishing Co., Berkely CA, 1965, p 943.

2. Magie, P. M., "A Review of the Properties and Potentials of the New Heavy Metal Derivative Solid Lubricants," <u>Lub Eng.</u>, 22, pp 262-263 (1966).

3. Campbell, M. E., "Solid Lubrication Technology: A Review", <u>Mech. Eng.</u>, 1968, p 28.

4. Centers, P. W., "The Role of Bulk Additives in Solid Lubricant Compacts," Ph.D Dissertation, University of Dayton, 1986, pp 202-204.

5. Quinn, T. F. J. and W. O. Winer, "The Thermal Aspects of Oxidative Wear," <u>WEAR</u>, <u>102</u>, (1985) 68-76.

6. Campbell, M. E., "Solid Lubricants, A Survey," NASA SP-5059(01), National Aerospace Adm., Washington DC, 1972, p 54.

7. Rabinowicz, E., "Friction and Wear of Materials," Wiley, New York NY, 1965, p 94.

8. Bowden, F. P. and D. Tabor, "Friction and Lubrication", Aberdeen Univ. Press, 1956, pp 44-46.

9. Capps, W., P. B. Macedo, B. O'Meara, and T. A. Litovitz, "Temperature Dependence of the High-Frequency Module of Vitreous B_2O_3," <u>J. Chem. Phy.</u>, Vol. 45, (1966) 3436.